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REMOVAL OF IRON CONTENT IN FELDSPAR ORES BY LEACHING WITH ORGANIC ACIDS

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Abstract: There are numerous physical and chemical separation methods for reducing iron content in feldspar ores. Generally, while -500+75 μm sized feldspar ores beneficiate by physical and physicochemical separation methods such as magnetic separation and flotation, -75 μm sized ones are discarded as slime sized tailings. In these research; oxalic, citric and glycolic acids which are principal organic acids to be used in leaching were used in these slime sized (-75 μm) feldspar ores in order to reduce iron amount. Temperature, pulp density, leaching time and acid molarity parameters were investigated by applying factorial design ANOVA-Yates test technique. As a result of the tests, effective parameters were determined and discussed. The best result was obtained as 67.90 % iron removal by using oxalic acid.

Keywords: leaching, feldspar, organic acids, statistical analysis.

INTRODUCTION

Feldspars are igneous aluminosilicate minerals with varying quantities of K, Na and Ca in their structure. The major feldspar minerals are K-feldspar (microcline), Na-feldspar (albite) and Ca-Feldspar (anorthite) (Doğu and Arol, 2004; Demir et al. 2001). Feldspars are used in glassmaking, ceramics, and to some extent as a filler and extender in paint, plastics, and rubber. Their chemical compositions; especially alkali contents determine their quality and price. In glassmaking, alumina from feldspar improves product hardness, durability, and resistance to chemical corrosion. In ceramics, feldspar (calcium oxide, potassium oxide, and sodium oxide) act as a flux, lowering the melting temperature of a mixture. Fluxes melt at an early stage in the firing process, forming a glassy matrix that bonds the other components of the system together. Of the domestic feldspar sold or used, an estimated two-thirds by tonnage went into the manufacture of glass, including glass containers and glass fiber (Tanner, 2012; Bayraktar and Çakır, 2002). It can be said that there is continuing increase in the feldspar production in the world when looking from the year 2005 (Figure 1). Turkey which exports about two-thirds of its production of feldspar is leader supplier of feldspar, currently. Feldspar mined in Anatolia in the south-western part of the country is mainly produced from the Region of Menderes Massif (Tanner, 2012). Feldspar ores often associated with the impurities like Fe

that decrease their economic value and deteriorate their other quality properties. Presence of iron in feldspar is not desirable because it changes colour for the final product, and therefore, iron content has to be kept below as a certain value (0.10% for glass making and 0.07 –0.08% for ceramics). A number of separation ways including chemical, physical and physicochemical methods are applied to feldspar ores in order to remove these dark-colored or undesired coloring impurities (Doğu and Arol, 2004; Styriakova et al, 2006; Çelik et al. 1998). Because feldspar ores concentrates in between 500 and 74 microns size naturally, slime sized ones are discarded to tailings in the industrial scale applications (Amarante et al, 1997). There are numerous studies involving feldspar ores beneficiation from magnetic separation to bioleaching. But flotation method is first choice when it comes to economy. Although the concentration scheme is usually dependent upon the quality of the end product, flotation is almost invariably the most widely used beneficiation method. While the floatability of albite using cationic reagents is maximum in the pH range of 8 to 9, feldspar-quartz separation is achieved only at low pH of about 2.5, using HF which suppresses quartz and activates feldspar. Mica is readily floated by an amine collector in a pulp pH of about 3. Iron-bearing minerals can be floated away either using sulfonate or fatty acid type collectors in acid circuit (Hacıfazlıoğlu et al., 2012; Gaied and Galala, 2011; Kangal and Güney, 2003; Çelik et al. 1998).

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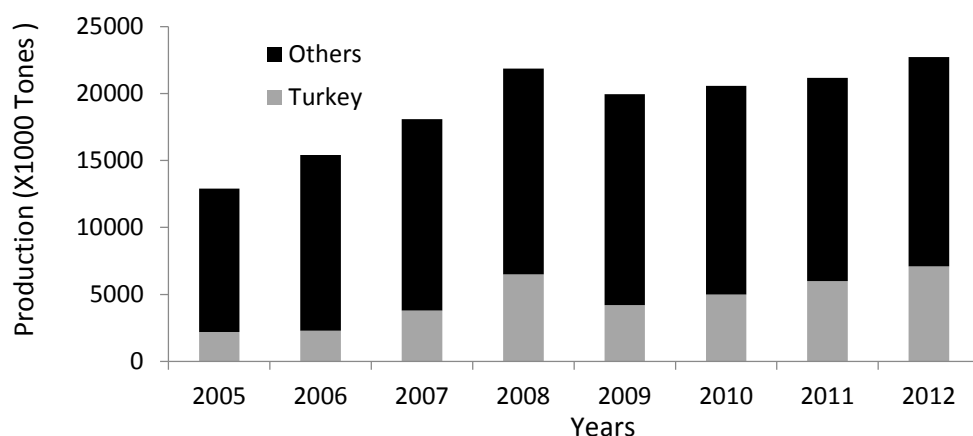


Figure 1, Production of Feldspar in the World (USGS, 2014).

Acid leaching for removing Fe from feldspar ores is an alternative way. Organic acids which have -COOH groups in their chemical formula, are less corrosive than inorganic ones, can be used to dissolve impurities. Moreover, they are biodegradable and can achieve a higher metal extraction efficiency at mildly acidic pH compared to other extracting agents (Jonglertjunya and Rubcumintara, 2013; Del Dacera and Babel, 2006; Bayat et al., 2004; Cameselle et al, 2003).

In these research; oxalic, citric and glycolic acids were used in order to reduce iron amount in feldspar ores. The leaching parameters were investigated by applying factorial design ANOVA-Yates test technique.

MATERIALS AND METHODS

Feldspar ores of Cam-Is Mining Company in which flotation and magnetic separation methods are applied were used as materials. Chemical analysis of the samples was performed by lithium tetraborate fusion followed by HCl dissolution and atomic absorption spectrophotometer (AAS) analysis, respectively. Wet chemical and size analysis results are shown in Table 1 and Figure 3. After comminution, materials were divided into three dimensional fractions as +500, -500+63 and -63 microns. Furthermore, iron oxide contents in different size fractions were determined by XRF results and graphed in Figure 2. Because most of the iron content were present in the slime sized fraction, it was decided that -75 microns sized samples were leached. It was detected that -75 microns sized samples contained 0.580% Fe₂O₃, 0.860% K₂O and 8.900% Na₂O according to the lithium tetraborate fusion

analysis. Besides, loss on ignition rate was 0.013%.

Table 1, Chemical Analysis of the Sample.

Content	grade%
SiO ₂	68.70
Al ₂ O ₃	18.00
Fe ₂ O ₃	0.254
TiO ₂	0.240
CaO	0.660
MgO	0.470
Na ₂ O	9.900
K ₂ O	0.380
P ₂ O ₅	0.170
Loss on ignition	0.113

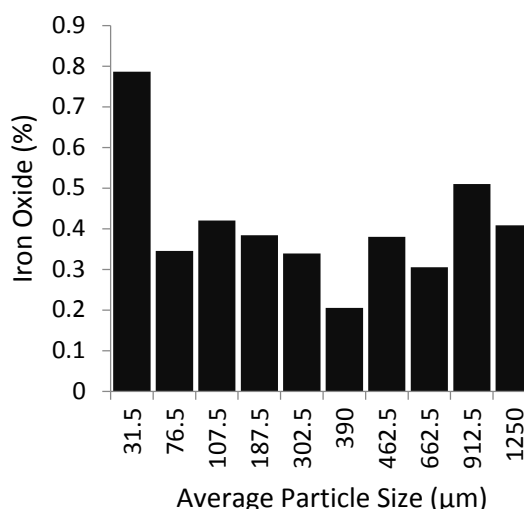


Figure 2, Fe Contents of the Fractions.

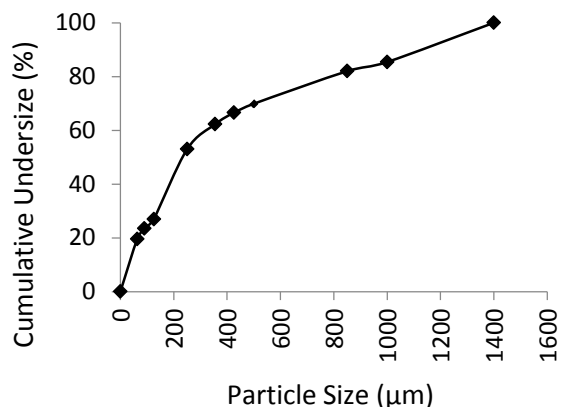


Figure 3, Cumulative Passing Undersize Curve for Original Feldspar Sample.

According to the XRD results, -75 μm sized samples mainly consist of Albite ($\text{NaAlSi}_3\text{O}_8$), Quartz (SiO_2), Muscovite ($\text{KAl}_2(\text{AlSi}_3\text{O}_{10})(\text{F},\text{OH})_2$) and Tourmaline ($((\text{Na},\text{Ca})(\text{Mg},\text{Li},\text{Al},\text{Fe}^{2+},\text{Fe}^{3+})_3(\text{Al},\text{Mg})_6(\text{BO}_3)_3\text{Si}_6\text{O}_{18}(\text{OH},\text{O},\text{F})_4)$ minerals (Figure 4).

The leaching tests were conducted in beaker which has 250 milliliters volume and the temperatures were controlled by thermometer. After the leaching was done, solid-liquid separation was made and iron extraction degree was detected by AAS for each test. Analytical grade chemicals and distilled water were used in the whole experimental study.

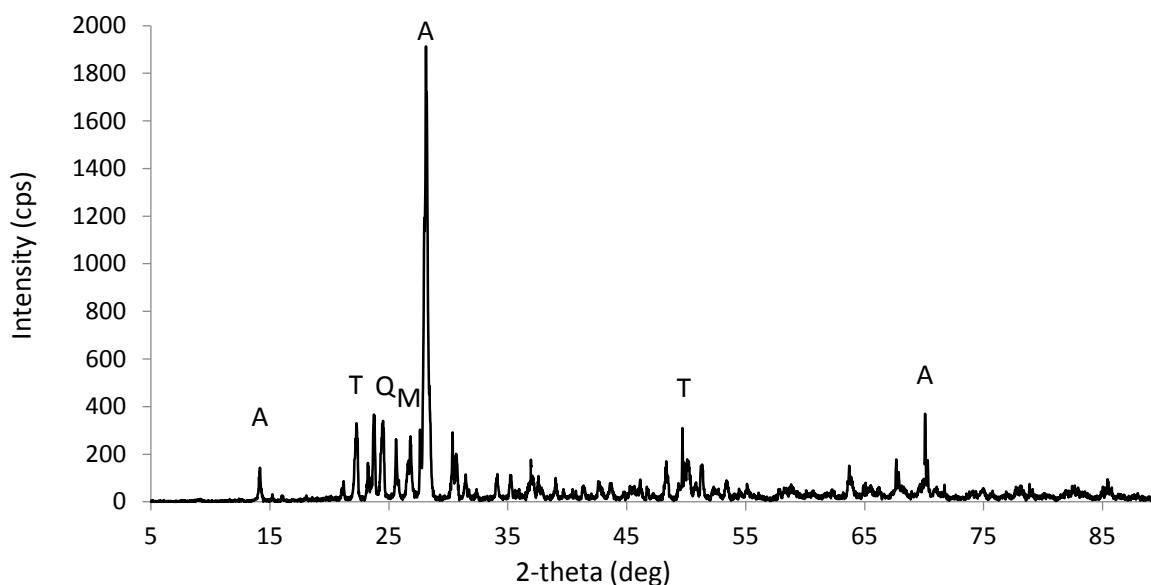


Figure 4, XRD Pattern of -75 Microns Sized Sample; Albite: (A), Quartz: (Q), Tourmaline: (T), Muscovite: (M).

The statistical design of experiments is used when it can be desired to learn the main and interaction effects of used experimental parameters. The effect of a variable is the change in response produced by varying the level of the factor. When the effect of a factor depends on the level of another factor, the two factors are said to interact (Çiftçi and Alandağ, 2014; Uçurum et al., 2006; Naik et al., 2005; Davies, 1993, Montgomery, 1991).

In this work, four variables were taken into consideration and the 2^4 full factorial unreplicated experiments were carried out in order to evaluate the main and interaction effects of them. Yates' notation has been used in this work to label each experiment. For example, treatment ABCD is the experimental run in

which the variables A (acid concentrate), B (solid/liquid ratio), C (time) and D (temperature) are set at their high level. Sixteen sets of trials were required according to the equation $N=2^n$, where N is number of trials and n is number of variables. Table 2 displays the tested treatment conditions and the corresponding experimental results obtained in the 16 tests (total 48 tests for 3 acids), in addition to the three replicated leaching tests, which constituted the central point of the experimental design, determined by arithmetic means of the high and low levels.. The higher level was designated as '+' and lower level as '-'. According to the basic principle of the design of experiments, four experiments were carried out at the base level to estimate error and standard deviation, where:

Y=response (%Fe extraction); X_1 =acid concentrate; X_2 = solid/liquid ratio, X_3 =time; X_4 =temperature. The ANOVA experimental parameter levels were chosen as follows: solid/liquid ratio, 10-30% (w/v); leaching time, 30-120 minutes; oxalic, gluconic and citric acid concentrations, 0.5-3.0 mol/l; temperature, 20-70 °C.

RESULTS AND DISCUSSION

The highest Fe extraction has been obtained at the AD combination (67.90%) by oxalic acid leaching, at the ACD combination (41.53%) by citric acid leaching and at the CD combination (34.61%) by gluconic acid leaching. It was

observed that average %Fe extraction in the leaching experiments conducted in triplicates in the central point was 44.80% with oxalic acid.

F values on the 9th column with "AB", "AC", "ABC", "AD", "BD", "ABD", "ACD", "ABCD" combinations obtained by the leaching experiments performed according to Yates design for Fe extraction were lower than the F table values given in the 10th column for oxalic acid. In this regard, it has been determined that these combinations are not active.

The regression equations with active and interactive terms can be written as seen in Equations [1], [2] and [3].

for oxalic acid:

$$y=32.56+3,030X_1-3,140X_2-14,067X_3+5,997X_2X_3+1,599X_4-6,417X_3X_4-2,201X_2X_3X_4 \quad [1]$$

for citric acid:

$$y=22,91+1,501X_1-0,876X_2+1,778X_3+1,519X_1X_3-0,692X_1X_2X_3+5,288X_4+1,895X_1X_4-1,551X_2X_4-0,342X_1X_2X_4+2,032X_3X_4-1,373X_2X_3X_4+0,286X_1X_2X_3X_4 \quad [2]$$

for gluconic acid:

$$y=21,16+1,386X_1+1,434X_1X_2+1,375X_3-0,703X_1X_3-0,553X_2X_3+0,703X_1X_2X_3+3,3377X_4-0,761X_2X_4-0,578X_1X_2X_4+1,365X_3X_4-0,633X_1X_3X_4-1,923X_2X_3X_4+0,953X_1X_2X_3X_4 \quad [3]$$

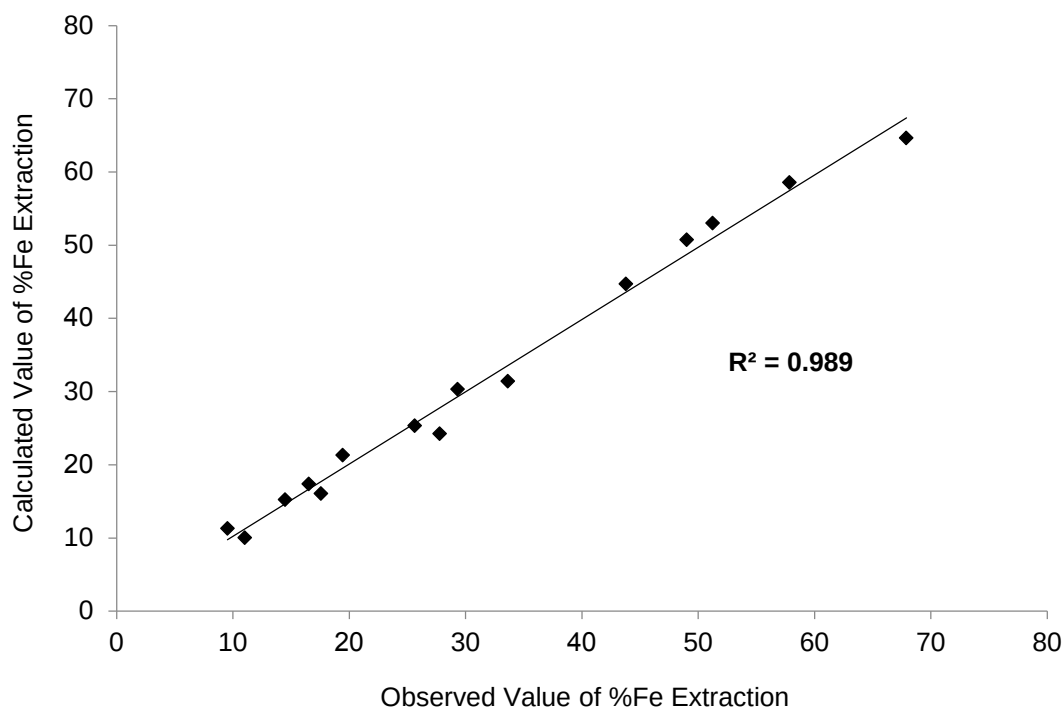


Figure 5, Fe Extraction Values Between Observed and Fitted Responses for Oxalic Acid Leaching.

Table 2, Oxalic Acid Leaching Experimental Results for Fe Removal from the Feldspar Ores*.

Yates Arrangement ^a	Experimental result (Fe extraction, %)	I ^b	II ^b	III ^b	IV ^b	MS ^c	DF _d	F(Calc.) ^e	F (Table) 1;16;0,0 05	Decision	X ₁	X ₂	X ₃	X ₄	Y (Calculation) ^f (Fe extraction, %)
1	46,09	97,34	154,43	247,66	520,92	569,40	1	95,789	4,49	Active	-1	-1	-1	-1	
a	51,25	57,09	93,23	273,25	48,48	146,92	1	24,715	4,49	Active	1	-1	-1	-1	52,985
b	27,78	33,95	218,56	19,67	-50,39	158,68	1	26,693	4,49	Active	-1	1	-1	-1	24,230
ab	29,31	59,28	54,69	28,81	-4,91	1,51	1	0,253	4,49	Inactive	1	1	-1	-1	30,291
c	14,50	125,74	6,70	-14,92	-225,08	3166,19	1	532,646	4,49	Active	-1	-1	1	-1	15,228
ac	19,45	92,82	12,97	-35,47	4,53	1,28	1	0,215	4,49	Inactive	1	-1	1	-1	21,289
bc	25,63	28,62	15,28	-0,55	95,95	575,43	1	96,804	4,49	Active	-1	1	1	-1	25,326
abc	33,65	26,07	13,54	-4,37	12,00	9,00	1	1,514	4,49	Inactive	1	1	1	-1	31,386
d	57,84	5,16	-40,25	-61,20	25,59	40,93	1	6,885	4,49	Active	-1	-1	-1	1	58,555
ad	67,90	1,54	25,33	-163,87	9,14	5,22	1	0,878	4,49	Inactive	1	-1	-1	1	64,616
bd	43,80	4,94	-32,92	6,27	-20,55	26,40	1	4,441	4,49	Inactive	-1	1	-1	1	44,665
abd	49,02	8,03	-2,55	-1,74	-3,82	0,91	1	0,153	4,49	Inactive	1	1	-1	1	50,725
cd	11,04	10,05	-3,63	65,58	-102,67	658,86	1	110,839	4,49	Active	-1	-1	1	1	9,995
acd	17,58	5,22	3,08	30,37	-8,01	4,01	1	0,673	4,49	Inactive	1	-1	1	1	16,055
bcd	9,53	6,54	-4,83	6,71	-35,22	77,51	1	13,039	4,49	Active	-1	1	1	1	11,289
abcd	16,54	7,00	0,46	5,29	-1,41	0,12	1	0,021	4,49	Inactive	1	1	1	1	17,349

^a a: acid concentrate (mol/lit), b: solid/liquid ratio(%), c: time (Minute) and d: temperature (C°)

^b Calculations of Yates algorithm for four parameters (n=4)

^c Mean square: (values of column IV)²/2

^d Degrees of freedom

^e F (Calculation): (MS/DF)*Variance (S²)

^f Y: Calculated value of %Fe extraction

*Arithmetic mean of Fe% extraction rate of the triple leaching tests performed at medium parameter values was 44.80%

CONCLUSIONS

This study demonstrates that the iron impurity in feldspar ores can be removed by organic acid leaching. The best result was obtained from the oxalic acid leaching. Because the organic acids are biodegradable and nature friendly, if a chemical process is necessary for removing iron from feldspars, organic acid leaching must be a good choice to reduce Fe contents of feldspar in glass making process. Moreover, minus 75 microns feldspar fraction which discharged into the tailings in the industry could be evaluated after overcoming furnace problems in glass and ceramic industry due to the low grain size.

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